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COMPLETE SPECIFICATION

Porous Materials Impregnated with Synthetic Resin and the Production Thereof

We, DYNAMIT ACTIEN GESELLSCHAFT VORMALS ALFRED NOBEL & Co., of Troisdorf Bez, Cologne, Germany, a joint stock company organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the impregnation of porous materials with synthetic resin.

It is known to impregnate porous materials, such for example as shaped bodies of graphite or carbon, with synthetic resins more especially for rendering them gas-tight and water-proof. To this end, the porous materials are impregnated with the hardenable liquid resin which is hardened after the impregnation. The hardening is generally effected by heating, usually under pressure.

It has also already been proposed to apply fluid pressure to the resin impregnated porous shaped bodies contained in a neutral medium in gaseous, vapour or liquid form in order to prevent the liquid resin from flowing or exuding from the porous bodies when heated to the hardening temperature. However, it has been found that it is not always possible to prevent the impregnating solution from flowing out despite the external pressure applied. Efforts have therefore been made to provide the impregnated shaped parts with a protective coating before the heat treatment, by applying a putty-like, self-hardening filling mass.

The present invention provides a process by means of which the impregnating liquid introduced in known manner into the pores of the materials can be hardened, without the application of heat, in such a manner as to penetrate deeply from the surface to the interior so that escape of the liquid from the pores is effectively prevented. According to the present invention a process for the impregnation of porous materials, such as shaped bodies of graphite or carbon with a hardenable synthetic resin and hardening the latter within the pores of the material, comprises impregnating the said materials with the hardenable synthetic resin in a liquid form and then submitting the impregnated material to the action of gaseous hardening agent which hardens the resin without the application of heat.

A hard shell is first formed on the surface pores of the material by the action of the gas. Preferably, the treatment is commenced with a hardening gas diluted with inert gas so that temperature increases due to the heat of reaction set up in the hardening can be avoided. After the formation of an outer shell, impermeable to liquid but permeable to gases, the gas concentration can be increased, since the resin mass within the workpieces which is still in the liquid state can then no longer escape. In the continuation of the treatment, the gas penetrates to a constantly increasing depth into the impregnated workpiece by diffusion, so that the depth of the hardening effect increases until finally the entire workpiece is hardened throughout. The diffusion may be promoted by alternate reductions and increases in the pressure of the gas employed in the treatment. The water forming during the hardening diffuses simultaneously to the surface of the workpiece and evaporates into the treatment gas. The moisture can be continuously extracted from the gas during the action by suitable drying means, so that the workpiece is not covered by a film of water which would prejudicially affect the final hardening process. The liquid resin employed may be a mixture of a synthetic resin with a solvent the viscosity of which mixture is lower than 1000 cP, and preferably 10 to 50 cP, the solvent being hardenable by the action of the hardening gas. For example, a mixture of a phenol-formaldehyde resin with furfurol as solvent has proved very suitable. Acid reacting gases may be employed with advantage for the

hardening, more especially halogens which react acid with the water of condensation or hydrogen halides.

The following examples illustrate the nature of the invention, and specific ways in which it may be carried into effect.

Examples.

1. Shaped graphite bodies to be impregnated are first degassed *in vacuo* in the impregnating vessel. To one part by weight of a phenolformaldehyde resin having a mol ratio aldehyde:phenol of 1:5 and a viscosity of 2000 cP, is added two parts by weight of furfurol. The viscosity of the mixture is about 10 cP. This mixture is drawn by suction into the impregnating container, whereafter the container may be placed under pressure. After 8 hours, the container is opened, the impregnating solution is drained off, and the bodies are removed and allowed to dry externally in the air. The impregnated bodies are then exposed to the action of hydrogen chloride gas in a gassing chamber. Gas diluted with air containing about 5% HCl is first employed and the concentration is slowly increased until finally, after about one day, the gassing is effected only with pure dry HCl gas. After two further days, the impregnating mass in the bodies has hardened and the bodies are gas-tight and liquid-tight. In tests it was found that even when the surfaces of the bodies treated as above are removed, e.g., by cutting or grinding, the bodies are proof against water at a pressure of 5 atmospheres gauge or against hydrogen under a pressure of 2 atmospheres gauge. The hardening period may be shortened by gradually increasing the gas pressure.

2. Porous bodies are degassed as in Example 1, impregnated with the liquid resin and dried. In the gassing chamber, they are exposed to the action of chlorine gas. By reason of the greater reactivity of chlorine, the gas concentration is initially made only about half as high as when hydrochloric acid is employed, that is to say, about 2-3%, in order to avoid an excessively violent reaction.

Instead of hydrogen chloride or chlorine gas, other acid-reacting or halogen gases may be employed, for example hydrogen bromide, or SO_2 (sulphur dioxide—sulphurous acid). An addition of materials having an accelerating action on the hardening process to the liquid impregnating medium is not necessary.

The process according to the invention is applicable with particular advantage to the production of shaped graphite bodies for chemical apparatus exposed to the action of highly injurious or corrosive liquids, gases or vapours, such as heat exchangers, cooling, condensation or absorption apparatus, distillation columns and the like.

What we claim is:

1. A process for the impregnation of porous materials, such as shaped bodies of graphite or carbon, with a hardenable synthetic resin and hardening the latter within the pores of the material, which process comprises impregnating the said materials with the hardenable synthetic resin in a liquid form and then submitting the impregnated material to the action of a gaseous hardening agent which hardens the resin without the application of heat.

2. A process as claimed in Claim 1, wherein the hardening synthetic resin employed is a phenolformaldehyde condensation product in the form of a solution in a solvent (for example, furfurol) which solvent is itself hardenable by the action of the gas, and the solution has a viscosity lower than 1000 cP (for example, 10-50 cP).

3. A process as claimed in Claim 1, or Claim 2, wherein the gaseous hardening agent employed is an acid reacting gas such as hydrogen chloride or chlorine.

4. A process as claimed in Claim 1, 2 or 3, wherein the concentration of the gas is increased during the hardening.

5. A process as claimed in any one of the preceding claims, wherein the pressure of the gas is increased during the hardening.

6. A process as claimed in any one of Claims 1 to 5, wherein moisture is extracted from the gaseous hardening agent during the hardening.

7. A process for the impregnation of porous materials, substantially is described in either of the specific examples herein.

8. Impregnated porous materials when prepared by the process claimed in any one of the preceding claims.

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